Fast Analysis of Low Levels of Dichloroethane using a Sample Concentrator and Varian CP-4900 DMD Micro-GC with Differential Mobility Detector

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Introduction

Detection levels in the low ppm range can generally be achieved for many components using the Varian CP-4900 Micro-GC and its thermal conductivity detector (TCD). However, detection levels can be improved by as much as 10X through the use of the Differential Mobility Detector (DMD) available on the Micro-GC. If a further improvement to detection levels is desired, more analyte must be presented to the instrument itself. The most common approach to achieve this is through the use of sample enrichment or concentration. The Enrichment and Desorption Unit (EDU) Sample Concentrator from Varian, Inc. is ideal for this purpose.

This note describes the analysis of very low levels (sub ppm) of 1,2-dichloroethane in which a special sample concentratior was used in conjunction with the Varian CP-4900 DMD Micro-GC with differential mobility detector.

1,2-Dichloroethane (EDC) is a colorless, oily, organic liquid with a sweet, chloroform-like odor. It is mainly used to produce vinyl chloride monomer, the major precursor for PVC production. It is also used as a solvent for resins and fats, and in photography, photocopying, cosmetics, drugs and as a fumigant for grains and orchards.

Recent government studies of volatile organic compounds have demonstrated a need for monitoring EDC in ambient air at the ppb level. The use of gas chromatography with Flame Ionization Detection (FID) for this type of analysis is quite straightforward. However, the sensitivity of FID is not sufficient to analyze ppb levels. The use of more selective detectors such as Electron Capture Detection (ECD) or Electrolytic Conductivity Detection (ELCD) is not particularly well suited due to lack of long-term stability and ease of use issues. DMD, however, offers both a high degree of sensitivity and selectivity.

Instrumentation

EDU-Varian Sample Concentrator with tenax adsorption material

Varian CP-4900 Micro-GC equipped with

- CP-Sil 5 CB column, 6 m
- Differential Mobility Detector (integrated)

GC control and datahandling software: Galaxie™ Software

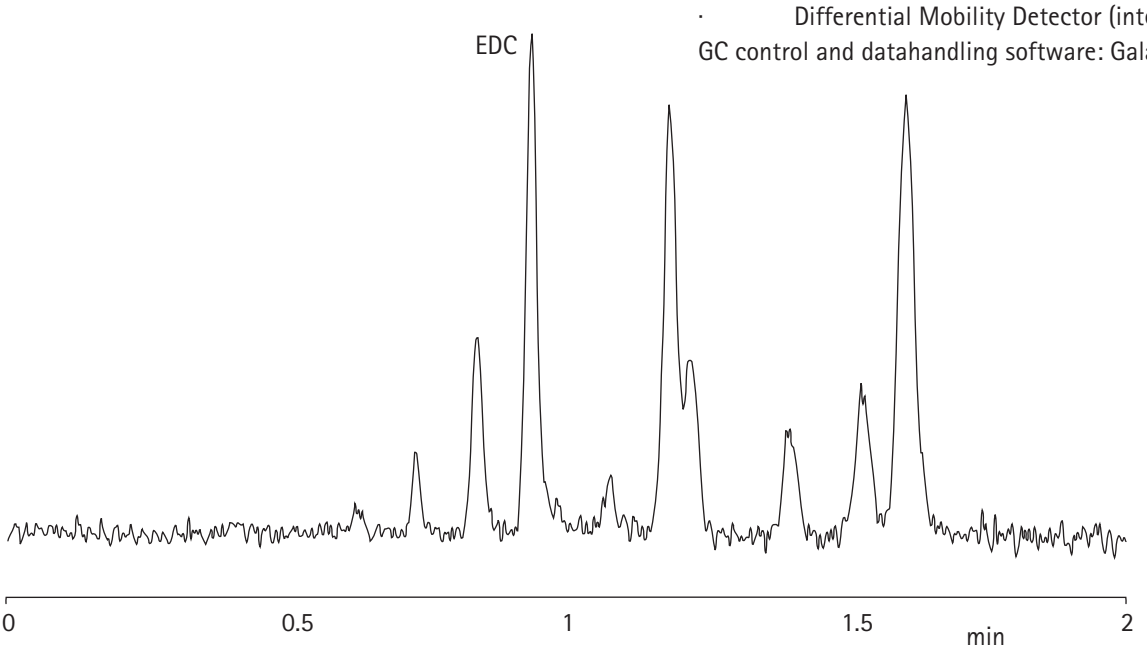

Figure 1. Chromatogram of a test sample containing approximately 3.5 ppb EDC.

Conditions

Settings Sample Concentrator

- Sampling 60 sec @ 30 °C
- Desorption 105 sec @ 200 °C
- Injection 15 sec @ 200 °C
- Cleaning 90 sec @ 225 °C
- Cooling 90 sec

Settings Differential Mobility Detector

- Vc EDC 27.59 V, negative channel
- Sensor temp. 80 °C
- Flow rate 450 mL/min, zero air
- Rf voltage 1200 V

Settings Varian CP-4900 Micro-GC

- Oven 100 °C
- Transfer temp. 50 °C
- Injection time 250 ms
- Injector temp. 50 °C
- Carrier gas He, 150 kPa
- Sample line temp. 50 °C
- Bunch rate 5 Hz

Results and Discussion

Using settings described above, a chromatogram of ~3.5 ppb of EDC was obtained (Figure 1). To further validate the system linearity and repeatability were tested. Linear behavior of sample concentration was demonstrated by increasing sampling time (Figure 2), giving a correlation coefficient of 0.9979.

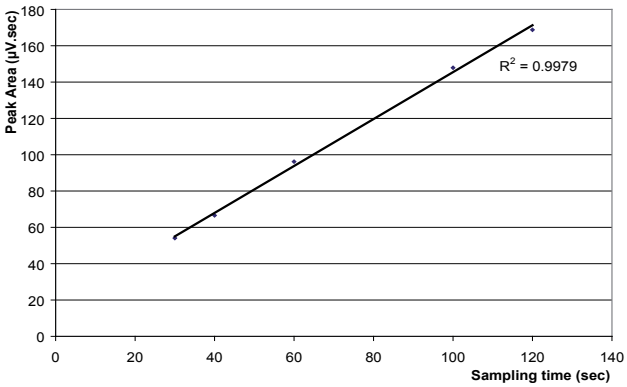

Figure 2. Linearity chart in sub ppb range.

To further check the system's integrity, run to run repeatability was tested. The air sample was run 14 times (Table 1). These results are shown graphically in Figure 3. The data presented in Table 1 and Figure 3 clearly show that even at low concentrations the results of 14 consecutive runs remain within a 10% RSD (grey area).

Table 1. Repeatability data

Run	Peak Area	Run	Peak Area
1	309.3	8	291.8
2	289.7	9	268.4
3	324.7	10	278.2
4	316.5	11	285.1
5	314.3	12	318.5
6	295.3	13	301.4
7	290.6	14	296.7
		Average	298.61
		St.Dev.	16.4
		RSD (%)	5.480

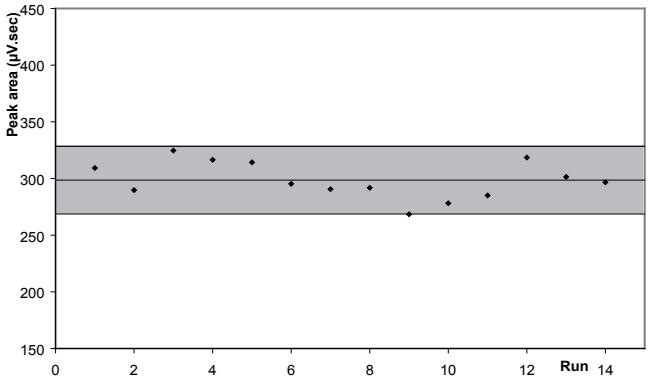

Figure 3. Repeatability data of 3.5 ppb EDC in air.

Conclusion

A dramatic improvement of detection limits in the analysis of dichloroethane in air can be achieved when sample concentration techniques are combined with a differential mobility detector equipped micro-GC. This two-fold increase is achieved first by the sample concentrator that increases the component amount in the sample, and secondly by the selectivity in combination with the sensitivity of the Differential Mobility Detector. This example clearly shows that by using the EDU-Varian Sample Concentrator and the Varian CP-4900 DMD Micro-GC with Differential Mobility Detector, low ppb concentrations of dichloroethane can be analyzed with very good repeatability.

Reference

Sutherland, F. *et al.* (2007) Determination of ultra trace levels of 1,2-dichloroethane in air by sample enrichement micromachined gas chromatography-differential mobility detection. Journal of Chromatographic Science vol 45, 8, 486-491.

These data represent typical results.
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